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A NUCLEONIC SENSOR FOR DETECTING METAL
IN RECIRCULATING LUBRICATING OIL SYSTEMS

Michael C. Bertin

Nucleonic Data Systems, Incorporated

Prepared for:

Army Air Mobility Research and Development
Laboratory

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NUCLEONIC SENSOR FOR DETECTING METAL IN RECIRCULATING LUBRICATING OIL SYSTEMS

By

Michael C. Bertin

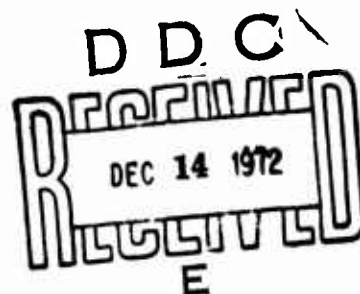
September 1972

**EUSTIS DIRECTORATE
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13. ABSTRACT This program was undertaken to determine the best way to detect metal contamination in lubricating oil. The goal was to detect contamination at the level of 10 parts per million. A nucleonic metal sensor (NMS) employing radioisotope-excited X-ray fluorescence technology has emerged as the solution to the problem. The NMS has detected both ferromagnetic and nonferromagnetic metals and has detected both metal particles and dissolved metals. Over the range 0 - 550 ppm metal concentration, the NMS has shown an accuracy of better than ± 6 ppm. Above 500 ppm, $\pm 1\%$ accuracy has been achieved.			

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This report was prepared by Nucleonic Data Systems, Inc., under the terms of Contract DAAJ02-71-C-0019. The work herein is part of a continuing effort to investigate and evaluate various techniques for determining the condition of aircraft components during flight.

The object of this program was to design, develop, fabricate, and test an in-line metallic debris sensor/monitor for recirculating lubricating oil systems, the rate of buildup of the metallic debris being a function of the degradation of oil-wetted components.

The design was based on X-ray fluorescence (XRF). In addition to detecting the level of contamination, XRF can identify the nature of the contamination by its characteristic X-rays. Nucleonic methods are not limited by particle size, and they can detect dissolved metals as well as particles.

The designed unit demonstrated the ability to monitor the amount and rate of change of the amount of debris in a circulating lubricating oil system.

The results of this effort indicate the feasibility of monitoring circulating oil in flight.

The technical monitor for this contract was Mr. Meyer B. Salomonsky, Aircraft Subsystems and Equipment Division.

I-C

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USAAMRDL Technical Report 72-38
September 1972

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Final Report

By

Michael C. Bertin

Prepared by

Nucleonic Data Systems, Inc.
Santa Ana, California

for

EUSTIS DIRECTORATE
U. S. ARMY AIR MOBILITY RESEARCH AND DEVELOPMENT LABORATORY
FORT EUSTIS, VIRGINIA

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FOREWORD

The work reported herein was performed under Contract DAAJ02-71-C-0019 and was authorized by DA Task 1F162203A43405.

The author thanks Mr. M. Salomonsky and Mr. B. Poteate of the Eustis Directorate for providing oil samples from actual helicopter gearboxes, and transmission system drawings. Technical guidance regarding engine wear and metal contamination of oil was furnished by the above-mentioned gentlemen. Drs. D. Donhoffer and K. Duftschmid of Nucleonic Data Systems made many valuable contributions to the program. Dr. W. Buchman is also acknowledged for his work on the X-ray absorption portion of the program.

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LIST OF SYMBOLS

A	constant determining XRF yield
D/AC	digital to analog converter
E	incident radiation energy, keV
F	figure of merit, $\Delta I/\Delta I_s$
I	XRF yield, beam intensity, or count rate
NMS	nucleonic metal sensor
p	weight concentration
ppm	parts per million
T	temperature, °F
V	voltage, volts
VFC	voltage to frequency converter
x	oil sample thickness, cm or in.
XRA	X-ray absorption
XRF	X-ray fluorescence
α	volume expansion coefficient
Δ	used to indicate a change, e.g., ΔT
μ	mass absorption coefficient, cm^2/gm
μg	micrograms
ρ	density, gm/cm^3
σ	standard deviation
V	volume

1.0 INTRODUCTION

1.1 Motivation and Statement of Problem

The quantity of metallic debris in engine lubricating oil is an important indication of the condition of oil-wetted engine parts. Component failure in a mechanical system is usually a three-stage process. The first stage is a gradual wear of the component in normal use. The second stage is accelerated component wear. The final stage is rapid deterioration, leading to failure. Impending engine trouble can be diagnosed by monitoring the rate of change of metal concentration in the oil. The value of a metal-sensing device in ground-based aircraft maintenance programs and on-line monitoring in flight is enormous.

The purpose of this program was to develop a sensor capable of detecting metal particles of various sizes and/or metal in solution down to the level of 10 parts per million (ppm) weight concentration. The sensor was to be capable of detecting both magnetic and non-magnetic metals. With respect to possible application in airborne systems, the sensor was to have detection capability over the temperature range from ambient to 400°F and was to be capable of withstanding vibratory environments.

1.2 Measurement Techniques

A variety of approaches can be applied to the problem of detecting metal in oil. These include filtering, optical, electromagnetic, and nucleonic techniques. Filtering does not lend itself to on-line applications, and it limits the flow of the lubricant unless a bypass system is included. Optical methods, including transmission and reflection, are sensitive to color changes in the oil caused by local heating effects. An optical sensor must combine both transmission and reflection techniques, and it cannot distinguish metals from other types of contaminants. The sensitivity of electromagnetic methods (capacitive, conductive, or magnetic) will vary greatly from metal to metal and will depend on the physical chemistry or ferromagnetic properties of the metal-oil matrix. Metals dissolved in oil would present great difficulty for electromagnetic methods.

The two nucleonic methods which can be applied to the problem are X-ray fluorescence (XRF) and X-ray absorption (XRA). XRF is potentially the most powerful

method available since, in addition to detecting the level of contamination, it can identify the nature of the contamination by its characteristic X-rays. Nucleonic methods are not limited by particle size and can detect dissolved metals as well as particles. The primary limitation of nucleonic methods is that they cannot sample very deep layers of oil. In practice, this is not a severe limitation since lubricants are often carried in pipelines of less than 1 inch diameter. Furthermore, in a circulating oil system the metal-oil mixture will usually be homogeneous, and, were this not true, a centrifuge or other type of collection device could be used.

1.3 Applications of Nucleonic Measurement Techniques

Nucleonic measuring techniques have been used for many years in aircraft. Dr. D. Wright, President of Nucleonic Data Systems, pioneered the development of a radioisotope gauge for continuous measuring of oil quantity in the tanks of the F104 and other Air Force fighter planes.⁽¹⁾ This gauge, qualified to MIL-O-38338A specification, used a radioactive Krypton 85 (Kr^{85}) source and a Geiger-Müller tube to detect the radiation. Other radioisotopes, such as Tritium (H^3) and Promethium 147 (Pm^{147}), have long been used in luminous safety devices in aircraft.

Both XRA and XRF are widely used in industrial monitoring and control systems. On-line gauging equipment using radioisotope sources is used to measure thickness and density of materials in the metals, paper, plastics, and other industries. Nucleonic devices have also been used to measure relative concentrations of elements in a mixture. The present problem falls into this category. Sulphur in oil⁽²⁾, silver and bromine in developing solution⁽³⁾, and heavy metals in ores⁽⁴⁾ have been detected by nucleonic means.

1.4 Program Summary

From a technical standpoint, nucleonic methods appeared to hold great promise for detecting metal in oil at the level of 10 ppm. From a hardware standpoint, nucleonic gauging equipment has flown in airplanes, helicopters, and manned and unmanned space probes. Nucleonic Data Systems undertook the present contract to prove the feasibility of a nucleonic metal sensor (NMS).

Both XRA and XRF were studied as potential solutions to the problem. The XRA program progressed rapidly in its initial stages. Changes in oil temperature, however, presented a serious obstacle to this approach and eventually led to the adoption of the XRF technique. Although XRA techniques proved not to be the answer to the NMS problem, a great deal of information was gained while studying XRA as a potential solution. This information is included in the appendix.

The XRF program involved the testing and selecting of radioactive sources and X-ray detectors, constructing an electronics unit, and testing of the NMS prototype. The test program conclusively demonstrated the feasibility of detecting metals in oil to an accuracy of ± 5 ppm (2 standard deviations) over the range 0 - 100 ppm concentration and ± 6 ppm over the range 100 - 500 ppm. At higher concentrations the accuracy of the NMS was estimated at $\pm 1\%$. The results of these studies show that a practical, on-line, continuously monitoring, nucleonic metal sensor based on X-ray fluorescence technology could be used to detect metal contamination in oil.

Chapter 2 of this report contains the theoretical discussions of X-ray fluorescence applied to the problem of detecting metal in oil. Chapter 3 details the experimental procedures and results of the XRF studies. Chapter 4 is a discussion of these results. Chapter 5 contains comments on possible future work on the nucleonic metal sensor program.

2.0 X-RAY FLUORESCENCE - THEORY

X-ray fluorescence (XRF) is the emission of characteristic X-rays by a medium when the medium is exposed to a source of radiation. A typical XRF arrangement is shown in Figure 1. In the present work, the exciting radiation has been either X-rays, gamma rays, or bremsstrahlung emitted by radioactive sources. Radioactive sources are preferred over X-ray tubes for use in the NMS because of their small size and weight, portability, ruggedness of construction, and freedom from maintenance requirements.

Both the nature and quantity of the emitting material can be determined using XRF. The energy of the emitted X-rays reveals what materials are present. This requires a detector which is capable of energy discrimination. The yield of fluorescent X-rays is given by

$$I = I_s \frac{p}{p + A(1-p)} \quad (1)$$

I_s is the yield for a thick metal sample, p is the weight fraction of metal in oil, and A is a constant which depends on the mass absorption coefficients of the metal and oil for the incident and fluorescent radiation.

The saturation yield, I_s , depends primarily on the geometry of the source, sample, and detector, and on the energy of the incident radiation. The yield is greatest when the incident energy, E , is just above the K absorption edge. I_s decreases as $1/E^3$ for higher energy radiation⁽⁹⁾.

The constant A is given by

$$A = \frac{\mu_{oi} + \mu_{of}}{\mu_{mi} + \mu_{mf}} \quad (2)$$

The subscripts o and m refer to the oil and metal respectively, while f and i refer to incident and fluorescent radiation. The major constituent of oil is carbon, with hydrogen and oxygen present to lesser extents. In calculating the mass absorption coefficients, the effects of the hydrogen and oxygen are averaged, and to a good approximation $\mu_{oil} = \mu_{carbon}$. Figure 2 shows the mass absorption coefficients (in cm^2/gm) of carbon and iron versus incident energy, E . Figure 3 shows the dimensionless constant A as a function of energy for titanium, iron, and zinc. The value of A decreases with increasing energy above the K edge. Referring to equation (1), it can be seen that the yield is maximized for the smallest values of A . This consideration dictates using a source whose radiation lies close to the K edge of the metals which contaminate the oil.

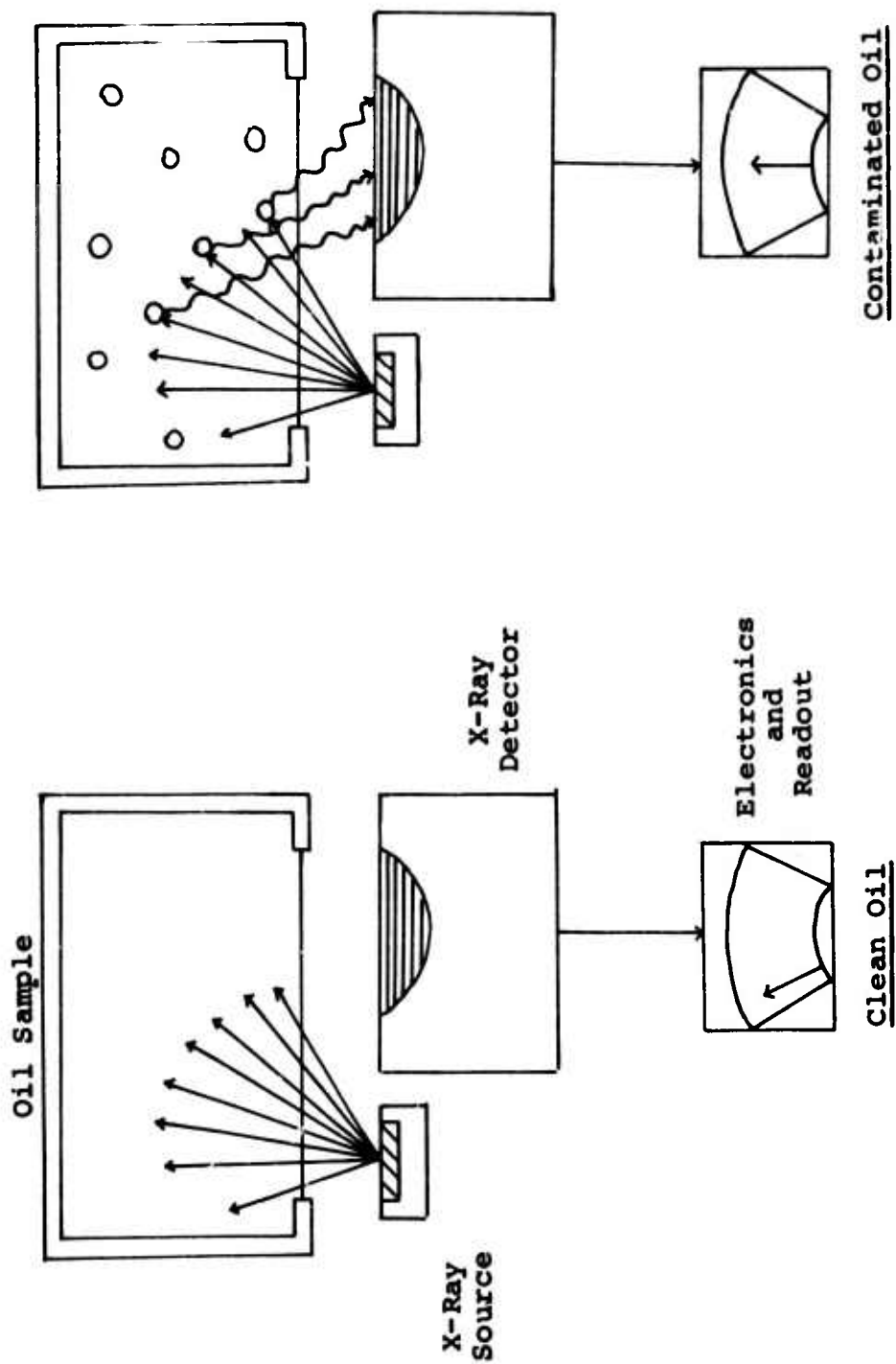


Figure 1. X-Ray Fluorescence System.

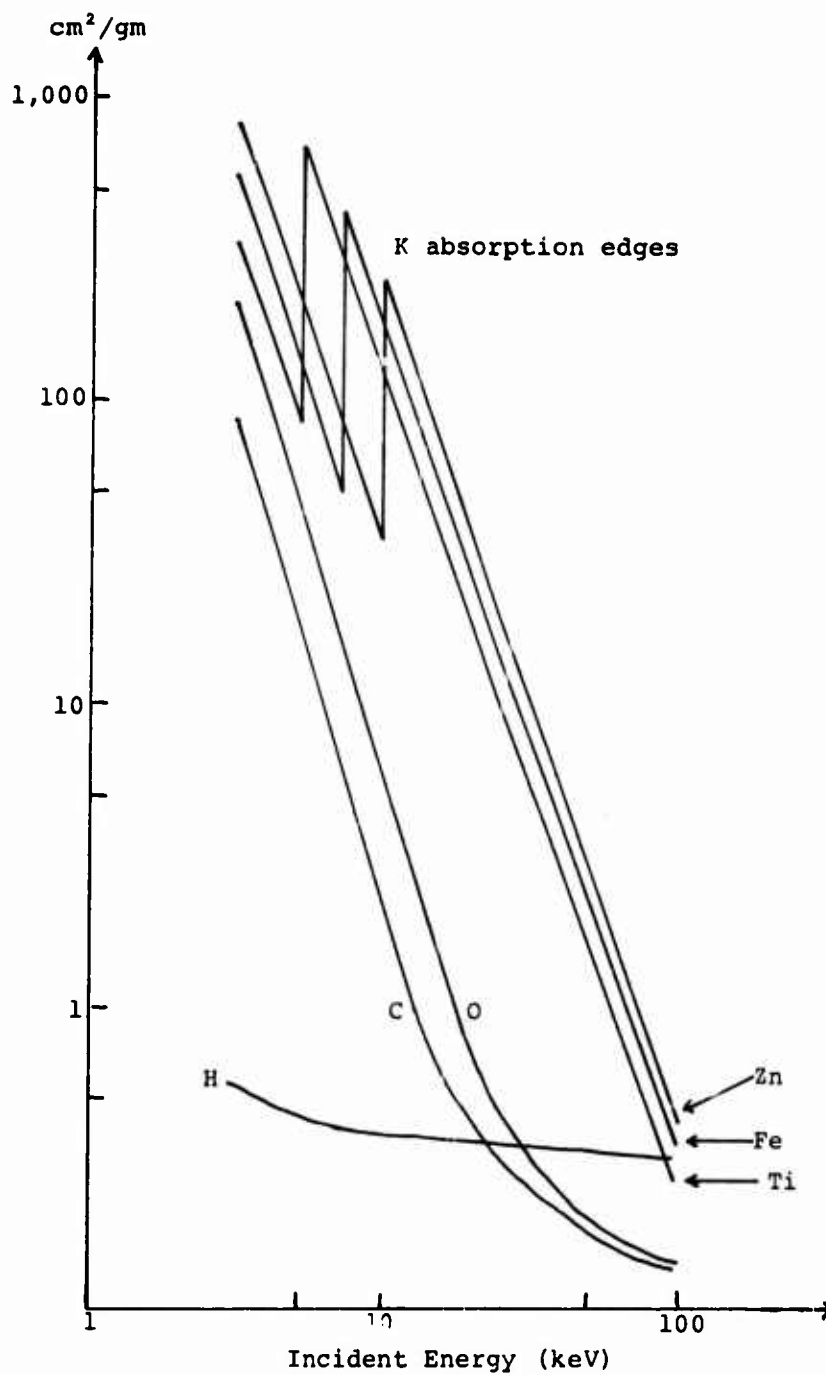


Figure 2. Mass Absorption Constant Versus Incident Energy.

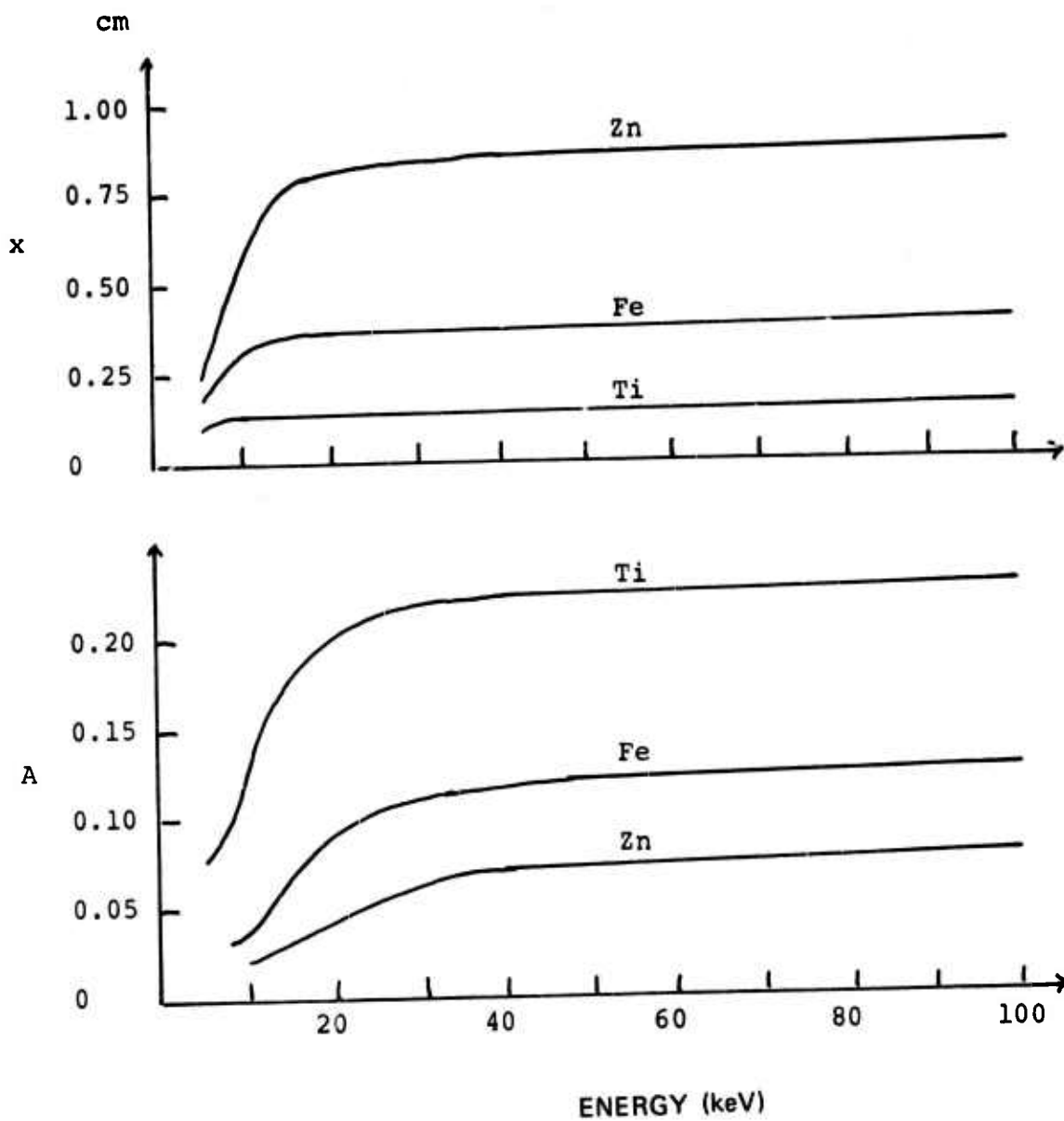


Figure 3. A and x vs Gamma Ray Energy.

The penetration depth of oil which can be viewed using XRF is also plotted in Figure 3, based on an oil density, ρ , of 0.95 gm/cm³. For $p < 10^{-3}$ (1000 ppm), this depth can be expressed as

$$x = 3 / (\mu_{oi} + \mu_{of}) \quad (3)$$

The XRF measuring technique should be insensitive to temperature changes. The main effect of temperature changes will be volume expansion of the oil, or a decrease in oil density. However, a decrease in oil density results in an increase in penetration depth, as seen from equation (3). In practice, there will be small yield variations with temperature due to heating in air gaps, expansion of detector windows, and electronics effects. These can be compensated for electronically.

Finally, equation (1) indicates that for metal concentrations less than 1000 ppm, the yield of fluorescent X-rays will be directly proportional to metal concentration.

3.0 EXPERIMENTAL PROCEDURES

3.1 Sources and Detectors

The major components of the nucleonic metal sensor are the source of incident radiation, X-ray detector, oil sample holder, and associated electronics package. The key to success in the NMS project proved to be selection of the right source and detector combination, subject to the limitation that the device be suitable for airborne applications.

The question of source selection is dependent on what metals one finds in the oil. The NMS has been designed to have maximum sensitivity in the region of atomic numbers 20 to 30, a range which includes titanium, iron, nickel, copper, and zinc. Purely technical considerations indicate the use of a source of 10 to 20 keV gamma or X-rays.

Practical considerations require the source to have a long half-life (i.e., greater than 10 years), be reasonably easy and economical to fabricate, and be radiologically safe for use in aircraft and ground-based maintenance programs. For airborne applications, a source with a biological half-life of less than 1 month is desirable (i.e., half of any material ingested is expelled from the body in less than 1 month). A monoenergetic source is preferable, though not essential.

Tables I and II compare the properties of primary and secondary X-ray sources. In a secondary source, X-rays are produced by another type of particle - a beta particle, for example. Only H^3 (tritium) and Kr^{85} (krypton) satisfy all the requirements. For ground-based equipment, short biological half-life is not as important a consideration and Pu^{238} (plutonium) could be used.

The detector used in an XRF system should have good energy resolution for X-rays and be capable of operating at elevated temperatures. This limits us to proportional counters. Avalanche detectors do not have sufficiently good resolution, and solid-state Si (Li) detectors or scintillation counters would not be suitable for a high-temperature environment.

End-window proportional counters proved to have poor energy resolution, and a side-window counter was selected. Figure 4 shows the response of argon-filled and krypton-filled proportional counters to the Pu^{238}

TABLE I. PROPERTIES OF NUCLIDES USED AS PRIMARY X-RAY SOURCES				
Nuclide	Half-Life (yr)	Mode of Decay	Photon Energy	
			keV	Biological Half-Life (yr)
Fe ⁵⁵	2.7	EC*	6 Mn K X-rays	2.2
As ⁷³	0.21	EC	10 Ge K X-rays	0.8
Pu ²³⁸	86.4	α	12 - 17 U L X-rays	200
Cd ¹⁰⁹	1.27	EC	88 22 Ag K X-rays	0.5
I ¹²⁵	0.16	EC	35 27 Te K X-rays	0.5
Pb ²¹⁰	22	β	47 11 - 13 Bi L X-rays plus bremsstrahlung up to 1.17 MeV	4
Am ²⁴¹	458	α	60 14 - 21 Np L X-rays 662	60
Gd ¹⁵³	0.65	EC	103 97 70 41 Eu K X-rays	1.5
Co ⁵⁷	0.74	EC	700 136 122 14 6.4 Fe K X-rays	0.03
*Electron capture.				

TABLE II. PROPERTIES OF NUCLIDES USED AS SECONDARY X-RAY SOURCES				
Nuclide	Half-Life (yr)	Mode of Decay	Useful Photon Energy Range (keV)	Biological Half-Life (keV)
H ³	12.3	β	4 - 12	0.03
Pm ¹⁴⁷	2.6	β	10 - 50 brem * 5 - 40 X-rays **	1.8
Kr ⁸⁵	10.7	β	25 - 80 brem 5 - 60 X-rays	<0.01
Sr ⁹⁰	28	β	50 - 200 brem	50
*bremsstrahlung				
**secondary X-rays				

source. The argon-filled detectors were decided upon because of the lower background in the region of the iron X-ray.

The initial proposal suggested the use of a Kr⁸⁵ beta source to produce secondary X-rays. This radioisotope is particularly suitable for airborne applications since it is an inert gas. It does not combine with body tissues and is expelled from the body in a matter of hours if ingested. In the event of an accident rupturing the source holder, the krypton gas is dispersed into the atmosphere where it is diluted to safe levels. A 514 keV gamma ray is emitted in 0.41% of the disintegrations, but this can be shielded against without unduly increasing the weight of the source. Krypton sources are currently in use in oil level gauges, in U.S. Air Force fighter planes and other aircraft.

The optimum geometrical configuration for the NMS calls for a transmission type Kr⁸⁵ source. This is shown in Figure 5. Beta particles from the krypton bombard the liner of the source, producing X-rays and bremsstrahlung. These are passed out through the front of the source. The beryllium window permits the X-rays and bremsstrahlung to pass, but keeps most of the betas in the source container. Germanium (10 keV X-rays) was tried as the liner, but fabrication of this liner proved to be difficult. Copper (8 keV X-rays) was used as an alternative. A 120-millicurie Kr⁸⁵ source was supplied for testing.

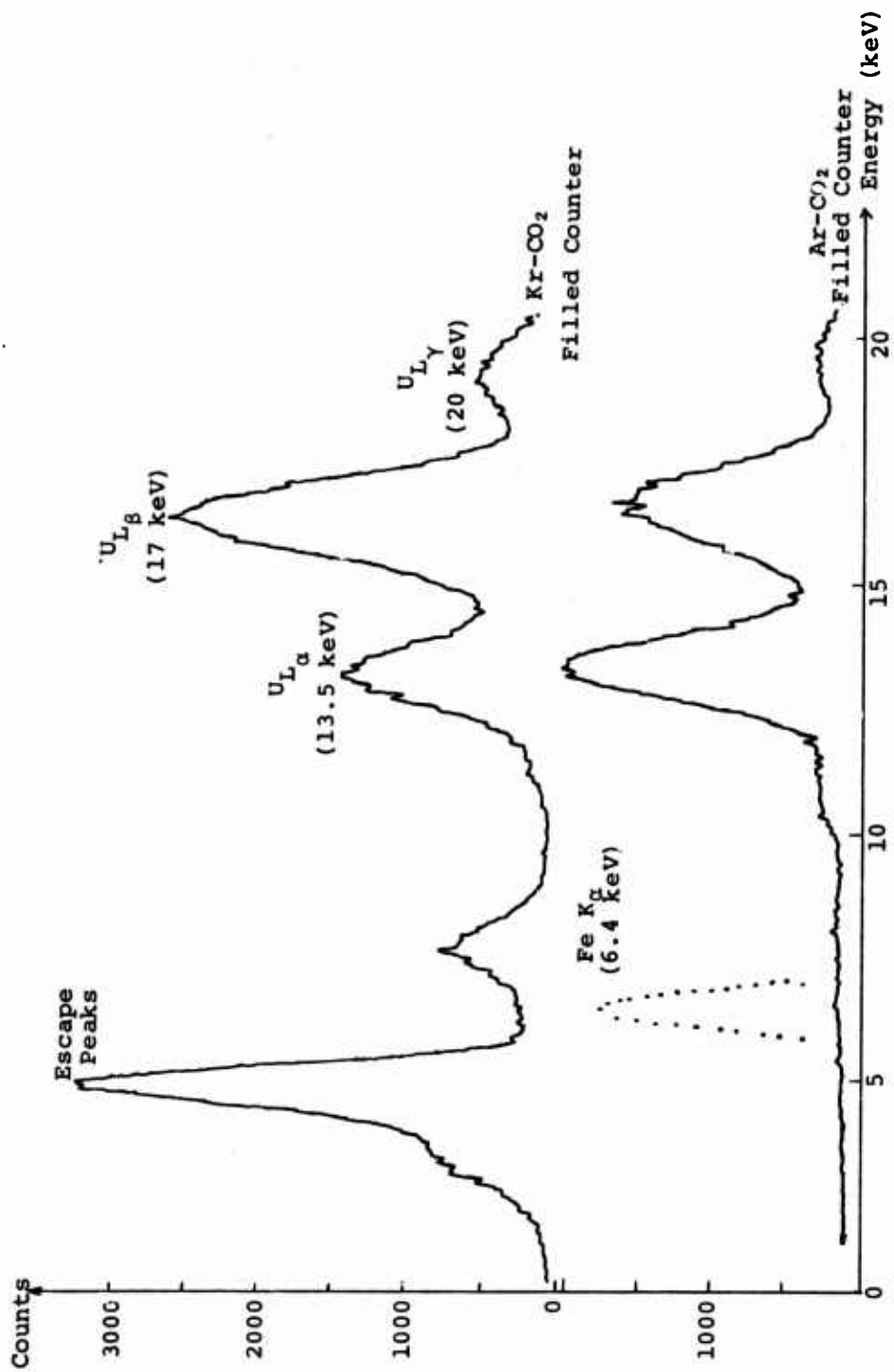


Figure 4. X-Ray Spectra for Pu^{238} Source in Argon- and Krypton-Filled Proportional Counters.

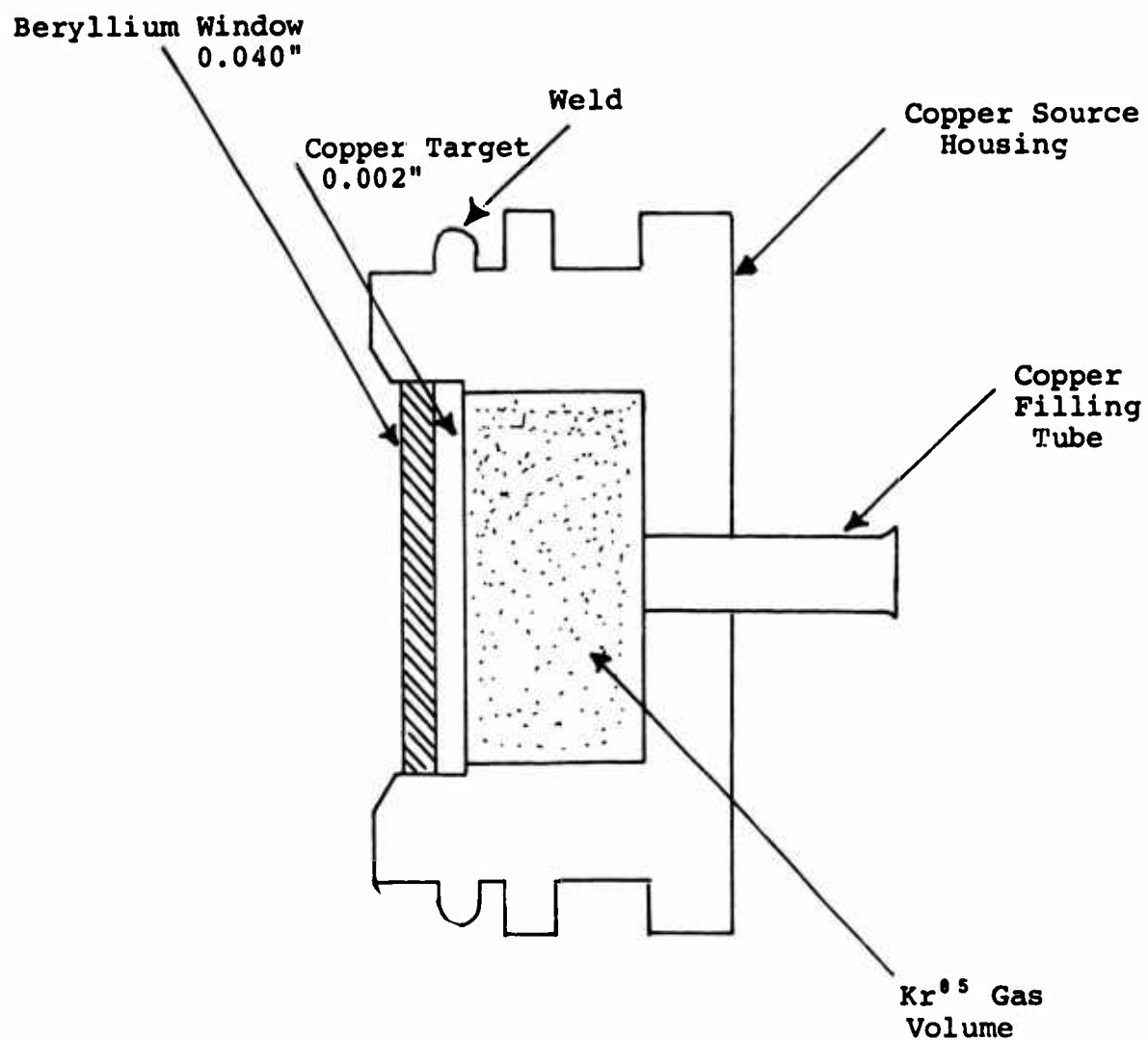


Figure 5. Kr^{85}/Cu X-Ray Source Design.

Tritium is also well suited to airborne applications because of its low-energy beta particles (maximum energy, 19 keV) and the fact that it dissolves easily in water. The radiological dose rate from tritium is very low since the betas are stopped by less than 1 centimeter of air. Ingested tritium is passed from the body in urine with a biological half-life of 12 days. Because of these facts, tritium in curie quantities has been widely used in luminous signs in aircraft. Solid tritium-zirconium bremsstrahlung sources, with a useful photon energy range of 5 to 10 keV, were selected for this project. A 16-curie source was supplied for testing.

A 10-millicurie Pu^{238} source was also obtained for testing. If the danger of an airplane crash is removed, as for ground-based equipment, plutonium becomes the most desirable source.

Figure 6 shows pulse height spectra for the three sources. The location of the iron X-ray is also shown on the figure. The uppermost spectrum is from the Kr^{85} source, and shows very clearly the copper X-ray from the source liner. The smaller peak at lower energy is the escape peak; the energy of this peak is the energy of the copper X-ray less 2 keV (the energy of an argon K X-ray). The middle spectrum shows the bremsstrahlung emitted by the tritium-zirconium source. All the beta particles emitted by the tritium are stopped in the source, and only the bremsstrahlung is used for fluorescence. The lower spectrum shows the uranium L X-rays emitted when Pu^{238} alpha decays to U^{234} . All the alpha particles are stopped in the source itself, and only the X-rays are emitted from the source capsule.

Figures 7 through 9 show pulse height spectra of scattered and fluorescent X-rays for clean oil and oil with 500 ppm of iron in solution.

Of the three sources tested, the Pu^{238} source had the best signal-to-noise ratio. The iron X-ray peak was clearly discernible down to 10 ppm. Figure 10 shows spectra for 0, 10, 20, 50, and 100 ppm. The number of counts in the region labelled "window" is listed in Table III for a 10-minute sample time. Figure 11 shows similar data for the tritium source. The large background is due to backscattering of tritium bremsstrahlung from the oil (Figure 6 shows the tritium source spectrum) and cannot be eliminated. The statistical counting error associated with the tritium source is about twice that from the Pu^{238} source for comparable geometry and counting time. Using Pu^{238} , the statistical error (1 standard deviation, σ) was ± 2 ppm below 100 ppm while for the tritium source the

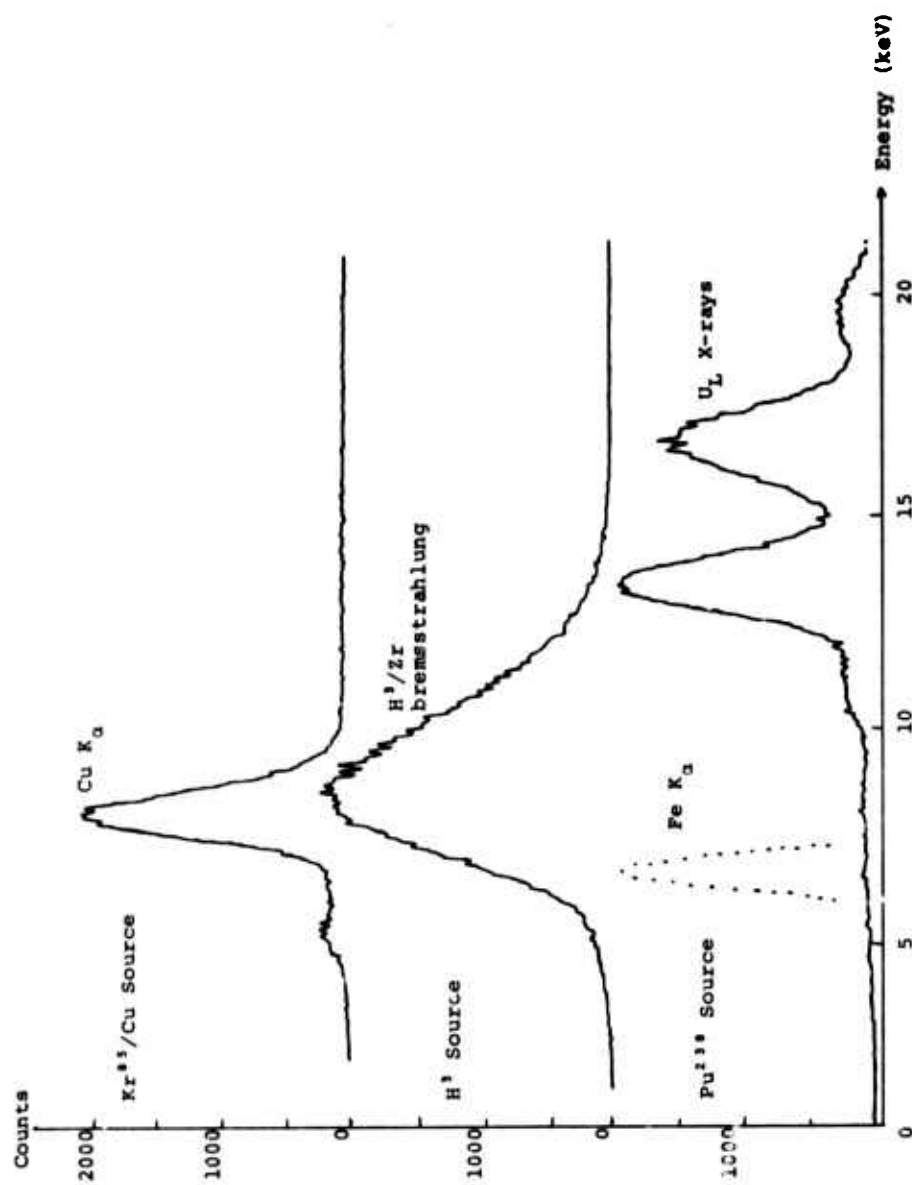


Figure 6. X-Ray Spectra - Pu²³⁹,
Kr⁸⁵/Cu, and H¹ Sources.

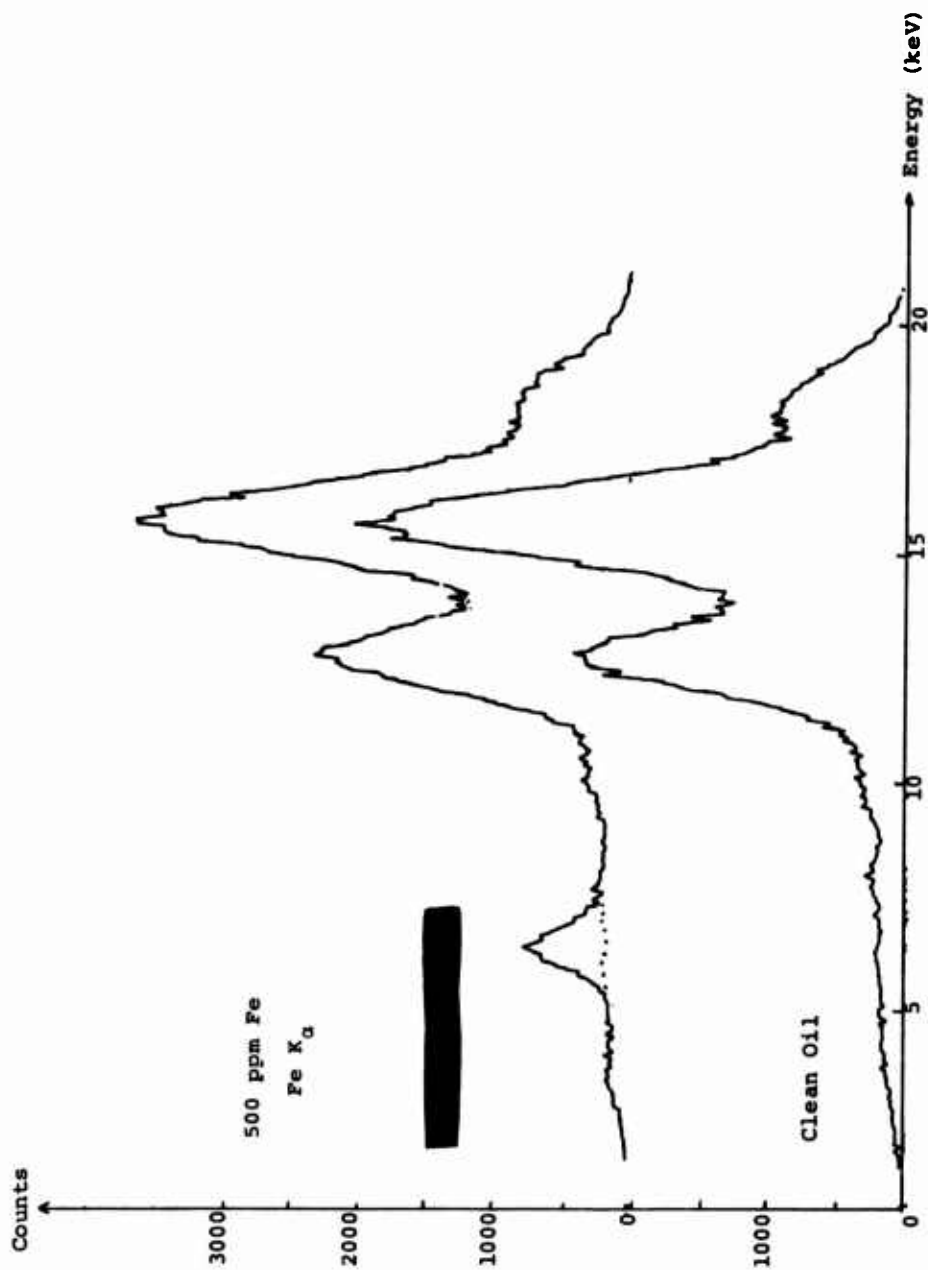


Figure 7. Fluorescent X-Ray Spectra,
Clean Oil and 500 ppm Fe - Pu²³⁸ Source.

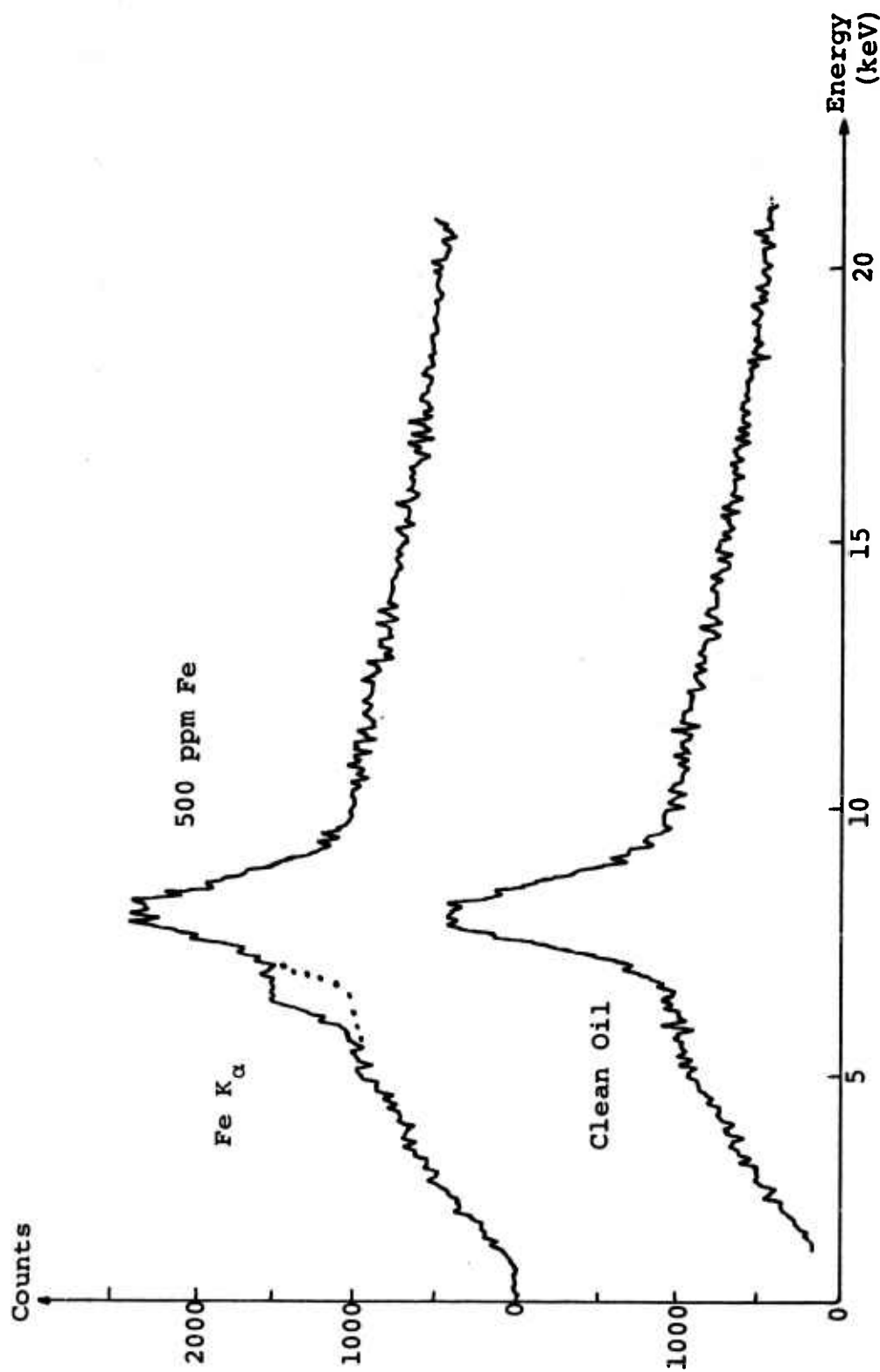


Figure 8. Fluorescent X-Ray Spectra, Clean Oil and 500 ppm Fe - Kr^{65}/Cu Source.

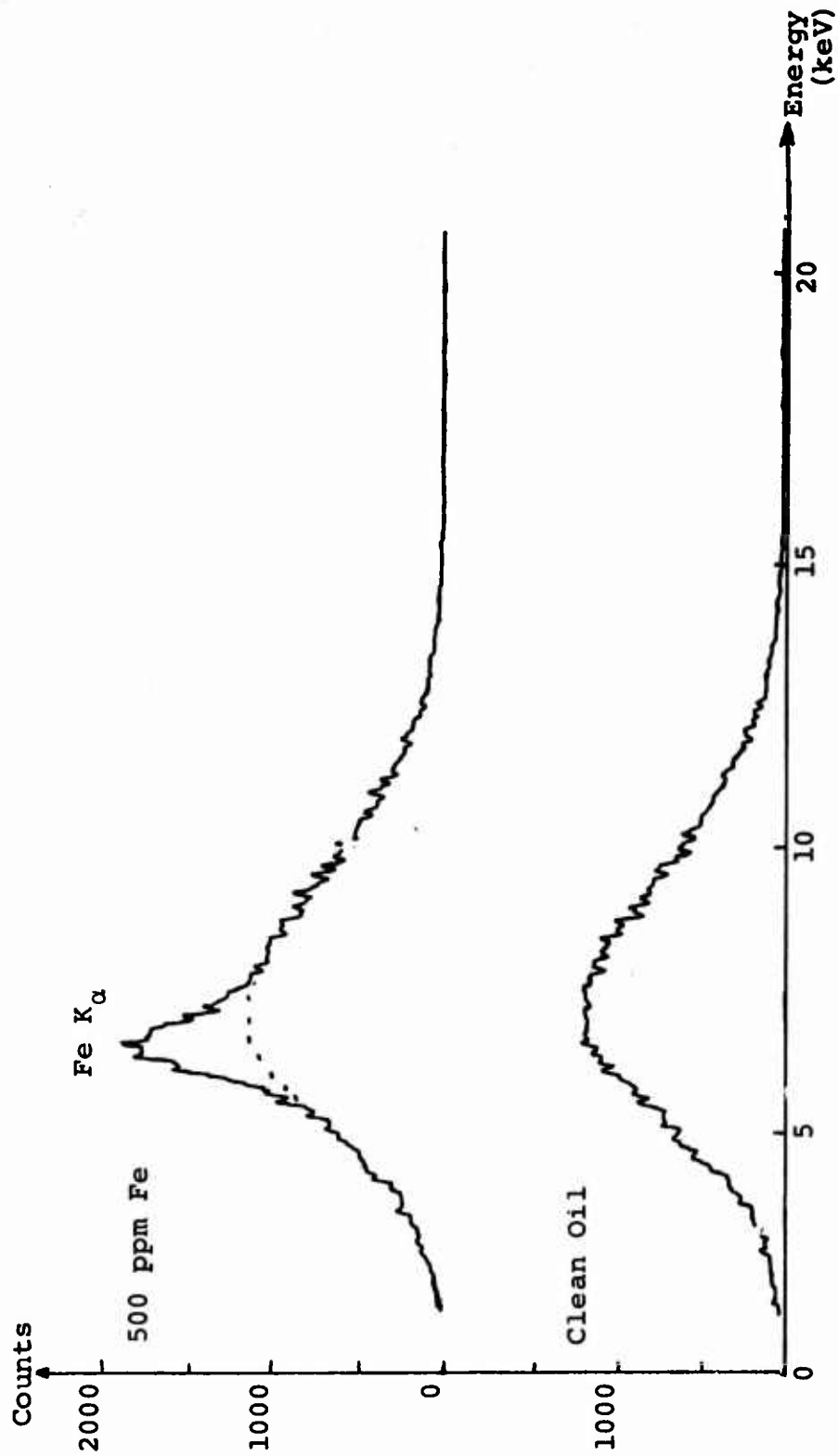


Figure 9. Fluorescent X-Ray Spectra,
Clean Oil and 500 ppm
Fe - H^3 Source.

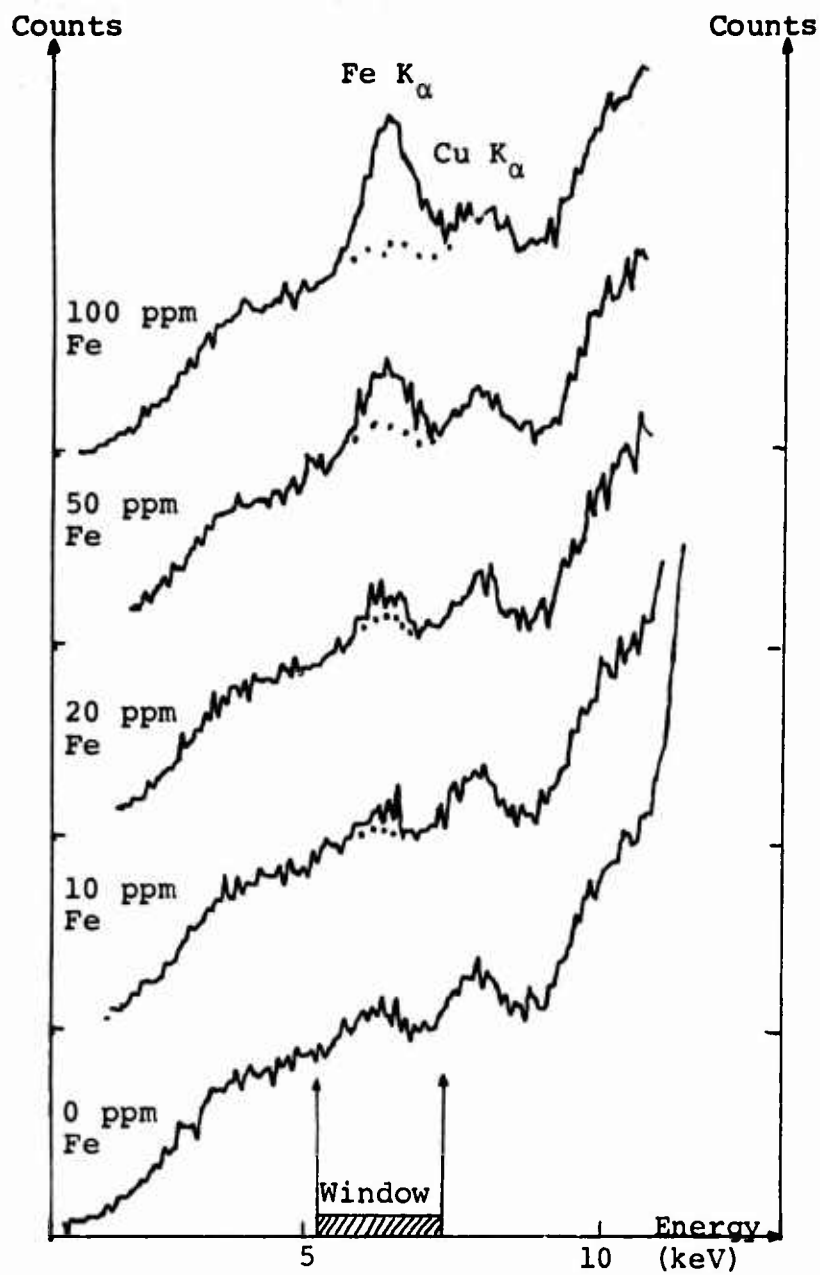


Figure 10. Various Concentrations of Iron - Pu^{238} Source.

TABLE III. COUNT RATE VS IRON CONCENTRATION
USING PLUTONIUM OR TRITIUM SOURCES

Iron Concen- tration (ppm)	Window Counts (10 min)		Net Counts		Statistical Error (1s) (ppm)	
	Pu ²³⁸	H ³	Pu ²³⁸	H ³	Pu ²³⁸	H ³
0	26,773	182,082	0	0	2.0	3.7
10	27,419	183,275	646	1,193	2.0	3.7
20	28,587	-	1,814	-	2.1	-
50	31,203	186,516	4,430	4,434	2.2	3.7
100	34,967	194,190	8,194	12,108	2.3	3.8
500	67,421	250,188	40,468	68,106	2.5	4.3

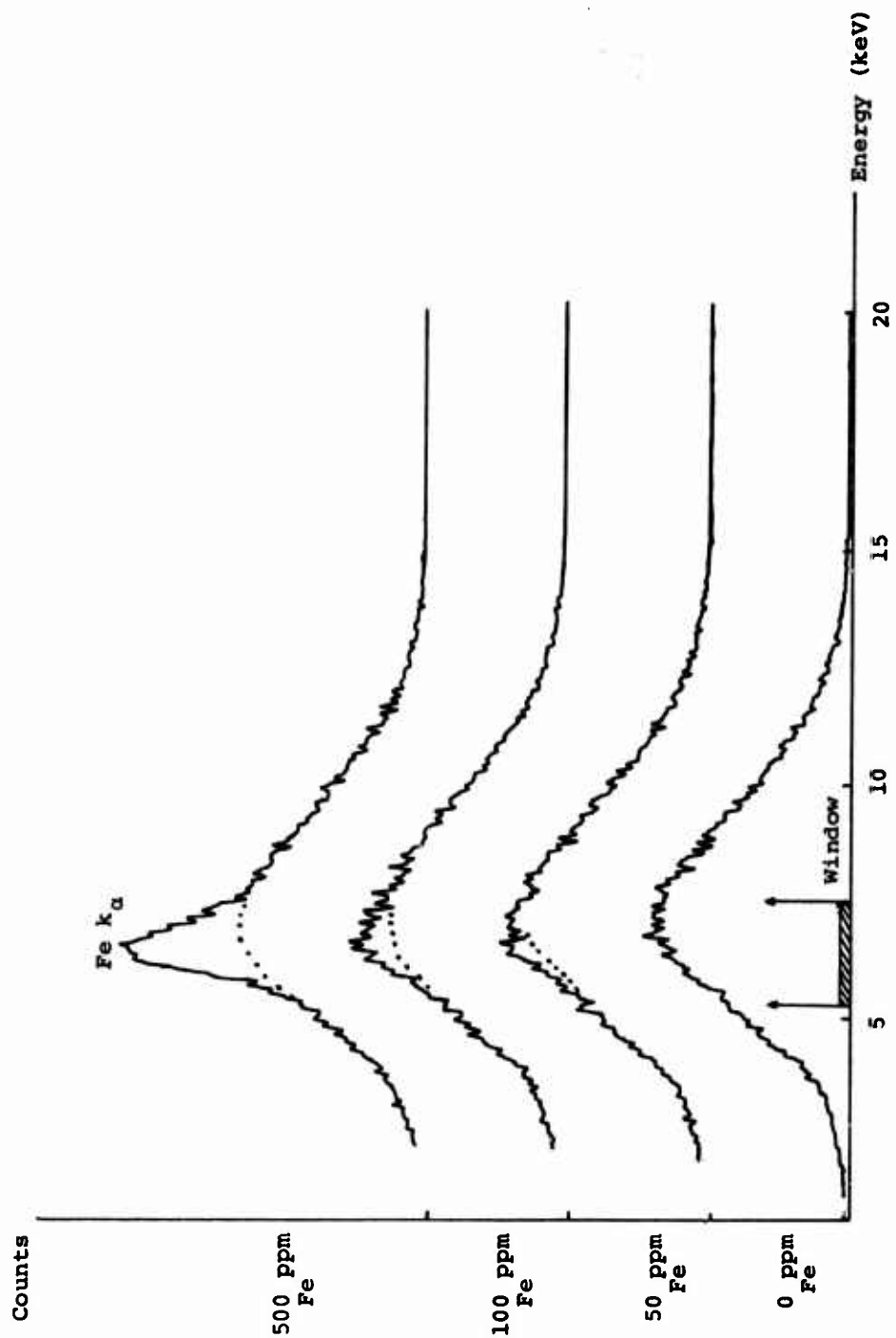


Figure 11. Various Concentrations
Of Iron - H β Source.

statistical error was ± 4 ppm.

The net count rate of iron X-rays produced by the Kr^{85} source was 2.2 times the count rate for the Pu^{238} source (compare Figures 7 and 8). The background in the vicinity of the iron X-ray, however, was approximately 20 times as large. This was caused by two effects - the use of copper as a source liner, and bremsstrahlung due to beta particles stopping in the beryllium window. A molybdenum liner should be used in future versions of this source, since molybdenum emits 17 keV X-rays, which would be well resolved from iron (6.4 keV). Secondly, a reflection type source should be used, rather than a transmission type.

The reflection type source would reduce the bremsstrahlung background. It was estimated that using the Kr^{85} source, the NMS would be as sensitive as with the Pu^{238} source. A new Kr^{85} source was not fabricated, however, for lack of time and money.

In view of these results, the Pu^{238} source was used to obtain the balance of the test data. For future programs a new Kr^{85} source should be designed, particularly for airborne applications. Experience with the present Kr^{85} source design indicates that this should be a relatively simple task.

3.2 Source and Detector Mounting

The source and detector mount in the NMS is extremely simple. The proportional counter is a stainless steel cylinder 2 inches in diameter. The source mounts in an aluminum ring which slips over the outer shell of the counter. This is shown in the upper portion of Figure 12. The lower portion of Figure 12 shows a method of mounting the NMS in a recirculating oil system. The drawing is a cross-sectional view through oil sample chamber. Oil would flow through this chamber in a direction perpendicular to the plane of the figure. The proportional counter tube and source, held by mounting rings and a mounting bracket, would be clamped or bolted to the oil sample chamber. A single high voltage cable connects the proportional counter to the electronics package. The actual test program did not employ a recirculating oil system. However, the results obtained with the XRF detection technique are independent of the flow rate of the oil.

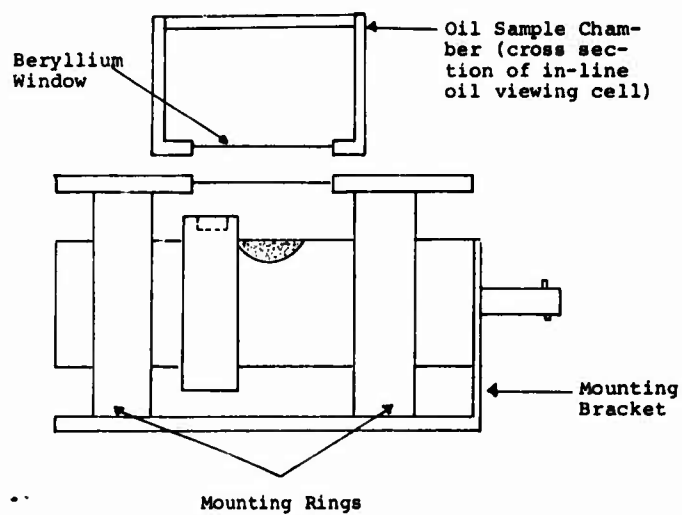
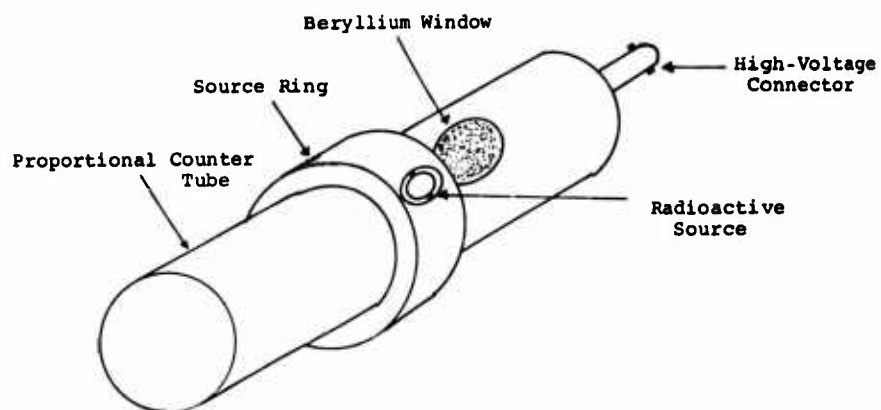


Figure 12. Source and Detector Mounting.

3.3 Oil Samples and Sample Holder

Because dissolved metals would not settle out or become inhomogeneous during testing, metal in solution was used for laboratory testing. Solutions would also be the best type of sample to use for calibrating the NMS. ENCO 2389 Turbo Oil was used in the testing program. This oil meets the MIL-L-7808 G specification.

Several organo-metallic compounds were tried in the preparation of oil samples. These included Tris (1-phenyl-1, 3-butanediono) iron, Tris (1-phenyl-1, 3-butanediono) chromium, ferric octoate, manganese octoate, and ferrocene. Ferrocene had the highest weight percentage of metal (30% iron) and proved to be the easiest to dissolve. Samples of a desired concentration were prepared by dissolving a weighed amount of ferrocene in a measured amount of oil. Other samples were prepared by dilution so that any concentration of metal in oil could be achieved. Some testing was also done with the octoate and "Tris" compounds; the results were similar to those observed with ferrocene.

In the early stages of the fluorescence program, thin iron foils were used to demonstrate the feasibility of the XRF technique. Two foils were obtained with surface densities of $98 \mu\text{g}/\text{cm}^2$ and $530 \mu\text{g}/\text{cm}^2$. When placed under clean oil samples these correspond to roughly 300 and 1500 ppm, respectively.

Tests were also performed with iron particles in oil, using particles ranging in size from 50 to 1000 microns. As expected, the NMS showed the same sensitivity to metal particles and dissolved metals.

The oil samples were tested in a container with a 1-mil-thick mylar bottom. The thin mylar window allowed the X-rays from the source to penetrate into the oil and fluorescent X-rays to reach the detector without being attenuated. The container itself was constructed from PVC pipe. When testing samples in aircraft, a beryllium window should be used instead of mylar. Beryllium metal is very light and has excellent strength properties. Beryllium windows are used in proportional counters and other radiation detectors. A 10-mil beryllium window would be extremely strong, but it would attenuate the iron X-rays by less than 10%. The 12-17 keV uranium X-rays would be attenuated by less than 2%.

3.4 Electronic Configuration

A block diagram of the electronics is shown in Figure 13. Oil is placed in the sample holder, and the sample is placed on the measuring head, which contains the radioactive source and proportional counter. A regulated high-voltage supply provides power to the proportional counter. Fluorescent X-rays from the oil sample are detected in the counter tube. The resulting signal is shaped and amplified in a preamplifier and postamplifier. The amplifier output then goes to a discriminator which generates a standard output pulse when the signal lies within a selected range of amplitudes. (This is the "window" region shown on Figures 10 and 11.) The discriminator pulses pass through a logic gate which is open for a specified time, and the pulses are accumulated in a scaler. At the end of the sample period, the contents of the scaler are placed in a memory register at the input to a digital-to-analog converter (D/AC). For this particular XRF application, a linear D/AC circuit was used since the number of counts accumulated in a sample period is a constant plus a number linearly proportional to the metal concentration (see equation (1) and Table III).

The output of the D/AC was used directly for the concentration display. Toward the end of the counting cycle, the output was sampled and held in an operational amplifier with capacitive feedback. At the completion of a new reading, the difference between the previous reading and the new reading was also displayed.

The gate between the discriminator and scaler is held open by a signal from the sample time generator. The concept here was to adjust for temperature changes, or other sources of count-rate changes, by increasing or decreasing the length of the sampling period. This is accomplished by using a voltage-to-frequency converter (VFC) and a time scaler. The output of the VFC is a pulse train whose frequency is proportional to the input voltage. By decreasing the input voltage, fewer pulses are generated by the VFC in a fixed time. The time scaler will be filled up when it receives a specified number of pulses from the VFC. When the input voltage at the VFC is low, the pulses arrive less frequently at the time scaler and the time scaler takes longer to fill. The gate remains open as long as the time scaler is not filled.

The NMS is designed so that the input voltage to the VFC is a constant voltage multiplied by a correction factor

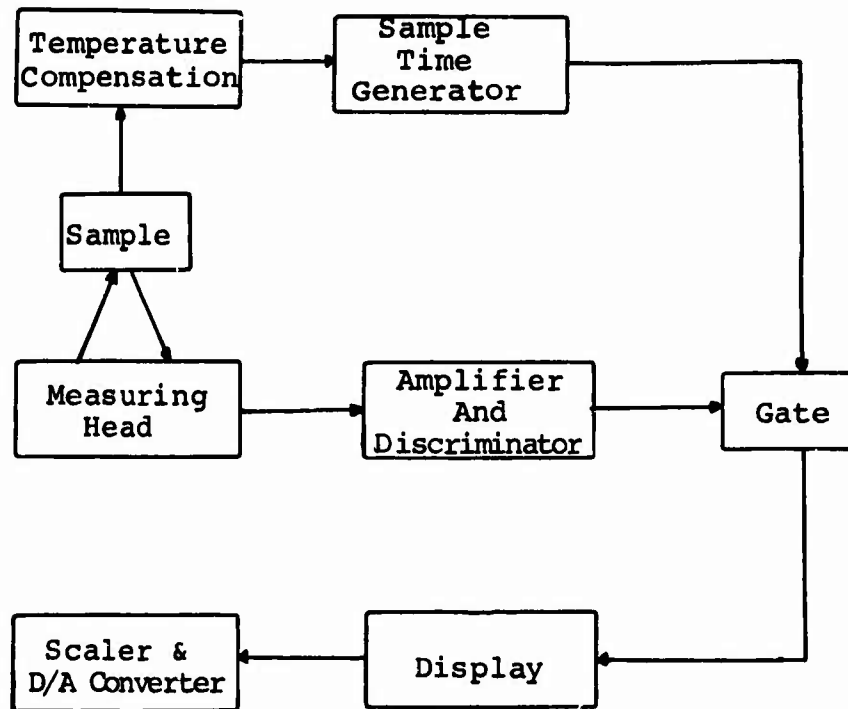


Figure 13. Electronic Configuration.

determined by the temperature. A platinum resistance thermometer measures the temperature of the oil sample, and the thermometer electronics converts the resistance change into a voltage which varies linearly with temperature. If V_t is the output of the thermometer electronics and V_o is the output of the temperature compensation circuit,

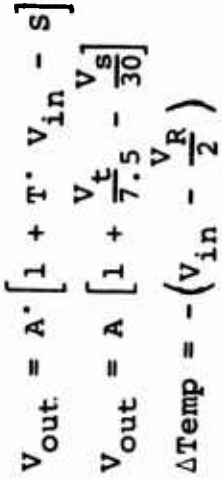
$$V_o = A(1 + b(V_t - c) - d)$$

The constants b , c , and d are adjustable voltages or potentiometer settings. The constant A represents the base time voltage which sets the counting time if " b " and " d " are set to zero. By connecting a variable input to the " d " parameter, this circuit can also compensate for changing count rates due to decay of the radioactive source. This was not necessary with the sources used in this project. Figure 14 shows the schematic of the temperature compensation circuit.

Much of the circuitry used in the NMS electronics package is proprietary to Nucleonic Data Systems and was previously developed with our own funds for commercial products. Only the temperature compensation circuit and sample and hold circuit were developed specifically for the NMS. The preamplifier, postamplifier, discriminator, gating, scaling, memory, D/AC, power supply, and timer circuits are standard NDS circuits or minor modifications thereof. The thermometer electronics, regulated high-voltage supply VFC, and ± 15 -volt power supply were purchased from commercial suppliers.

For this portion of the NMS program, an analog meter display was considered most appropriate. The meter giving the contamination level display is operated directly off the D/AC. Two potentiometers on the front panel provide adjustments for the zero and gain of the analog output. When a new measurement is completed, the digital information is fed to the D/AC and the meter updates immediately.

To provide contamination change information, it is necessary to store the previous reading for difference taking. The way this is done is shown in Figure 15. When the relay closes, the output voltage of the first operational amplifier becomes proportional to the D/AC voltage. When the relay opens, the feedback capacitor keeps that voltage constant even though the D/AC output may have changed. The operational amplifier used has a low input current so the capacitor is not discharged. The same



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input voltage is supplied to the second operational amplifier. The second amplifier takes the difference between the present D/AC valve and the valve stored on the first amplifier. Thus, the output of the second amplifier is proportional to the change in contamination.

The timing of the relay closure comes from the timer circuit. The two gates shown on Figure 15 provide the proper coincidence toward the end of the measurement cycle so that the current D/A output is stored just before completion of the new measurement cycle. The change reads zero during this brief period.

Permanent storage devices were investigated early in the program. Electromechanical counters with output switches were used in a feedback scheme. This system was made to work during preliminary tests, but it was not included in the final NMS electronics package.

3.5 Gauge Setup and Operation

The location and function of the NMS controls are shown in Table IV. The proportional counter is connected to the preamplifier by a single, high-voltage cable, and the gauge is ready for operation when plugged in and turned on. The high-voltage, baseline, and window potentiometers are set in the laboratory using a multichannel pulse height analyzer and should not be adjusted in the field. The window can be set to detect any element in the range from sulphur to germanium. For the present test program, it was set to detect iron, cobalt, nickel, and copper.

The sampling time is controlled by four potentiometers on the circuit board for temperature compensation. This is also set in the laboratory and should not be adjusted in the field. Two ranges of sampling time may be selected by means of the front panel switch: a long sample time and a short sample time. The short sample time is 1/16 of the long time. On the present NMS, these are set for 12 minutes and 45 seconds respectively. This could be reduced to 2 or 3 minutes if a stronger source were used.

The gain and zero controls are used in calibrating the NMS and need not be adjusted once the unit is calibrated. The zero potentiometer is set to make the meter read zero for clean oil. The gain potentiometer is then set so that full-scale deflection of the meter corresponds to the desired contamination range. In these tests the NMS was set to read 100 ppm full scale.

TABLE IV. NMS CONTROLS		
Control Name	Location and Description	Function
HV Adjust	Access through hole in front panel; potentiometer adjustable by screwdriver	Sets high voltage for proportional counter
Baseline (B)	Rear panel - 10 turn potentiometer	Sets lower level of window region
Window (W)	Rear panel - 10 turn potentiometer	Sets width of window region
Gain (G)	Front panel - 10 turn potentiometer	Sets gain (sensitivity) of NMS
Zero (Z)	Front panel - 10 turn potentiometer	Sets zero of NMS scale
Sample Time	Front panel - switch	Sets normal or short sample time (short = normal/16)
Sample Switch	Front panel - switch	Selects manual (single reading) or automatic (continuous reading)
Test Switch	Timer board - switch	Normally in "short", position; functions only when sample time is set to "short" Setting this switch to "test" position gives a reading in 1/16 the time of the "short" sample time
Switch Box	Rear panel - 12 switch control	Used for testing operation of D/AC and calibrating external recorders All switches normally open

Readout of the NMS is visual. The left-hand meter reads metal concentration directly in ppm. The right-hand meter reads change of concentration directly in ppm. The change of concentration meter is a center-reading meter and can indicate either positive or negative changes. A test point is provided on the sample and hold board for connecting the NMS to an external recorder.

Several test switches are provided at the rear of the unit to insure that the D/AC circuit is functioning correctly. A two-position switch is located on the timer board. The positions are marked "short" and "test", and the switch should be normally in the "short" position. When testing the D/AC circuit, put the sample time switch in the short sample time position and the test switch in the "test" position. This will cause the unit to read in 1/16 of the short sample time, or, roughly, every 3 seconds. A set of 10 switches is located on the black switch box at the rear of the electronics unit. The first 10 correspond to the 10 bits of the memory register at the input to the D/AC. Closing a switch corresponds to setting one of these bits. The switch box is useful for calibrating external recorders. In normal operation, all the switches should be open.

Field operation of the NMS is particularly simple. Set the sample switch to manual. Plug the unit in and turn it on. Select the long or short sample time. For a single reading, set the sample switch to "Auto", then return it immediately to "Manual". The readout will be completed automatically. For continuous readings, leave the sample switch on "Auto". Remember that the concentration change display drops to zero just before the completion of each reading.

3.6 Data Accumulation

Oil samples were prepared using the techniques described previously in strengths ranging from 10 to 500 ppm. The window was set using the thin iron foils. After adjusting the source position for maximum count rate, a preliminary calibration was made using the 45-second counting time. Final calibrations were made with the 12-minute counting time. Using the longer counting time reduces the statistical fluctuations by a factor of four, but it makes testing extremely time consuming.

With the gauge calibrated, readings were taken to determine the sensitivity of the NMS. The great majority of data was taken with samples in the range 0-100 ppm.

Some data was also taken at 500 ppm. Figure 16 shows an example of data accumulated during a 1-week period with the NMS. The samples were ferrocene dissolved in oil. The number of 12-minute readings is shown beside each point. The points represent the mean value of the readings, while the error bars represent two standard deviations. Also shown on the figure are distributions of readings (see box on Figure 16). It can be seen from the figure that the range of readings is typically ± 5 ppm; this corresponds to two standard deviations. The same conclusions hold up to 500 ppm. Table V contains the data from which Figure 16 was made. Data accumulated on a different occasion at 500 ppm had a two-standard-deviation error of ± 6 ppm.

Data accumulated with the 45-second sample time showed larger statistical fluctuations. Table VI shows data taken and normalized to the same calibration. Statistical fluctuations are on the order of ± 12 ppm (two standard deviations).

Tests were also made with iron and copper particles in oil. The NMS responded with excellent sensitivity to particles. Calibrated solutions of metal particles in oil were not available, however, and the NMS could not be checked against any known standard. It would be of value to have calibrated samples from other U. S. Army programs to check the NMS response. This would require installing the NMS on a circulating oil sample holder to prevent settling out of the particles.

3.7 Additional Tests

Data was also accumulated for oil samples containing copper particles and copper-iron particle mixtures. The NMS was able to resolve copper from iron and to detect either copper or iron, or both simultaneously, down to the level of 10 ppm. A copper X-ray from a contaminant corresponding to roughly 30 ppm is shown in Figure 10.

Samples of actual helicopter transmission oil from Fort Eustis were supplied and tested. Contamination levels ranged from zero to 17 ppm. Some of these samples had minute particles held in suspension.

The components of the measuring head were tested for performance at elevated temperatures. The proportional counter and Pu^{238} source were set up to fluoresce a thin iron foil and heated to 300°F. The proportional counter

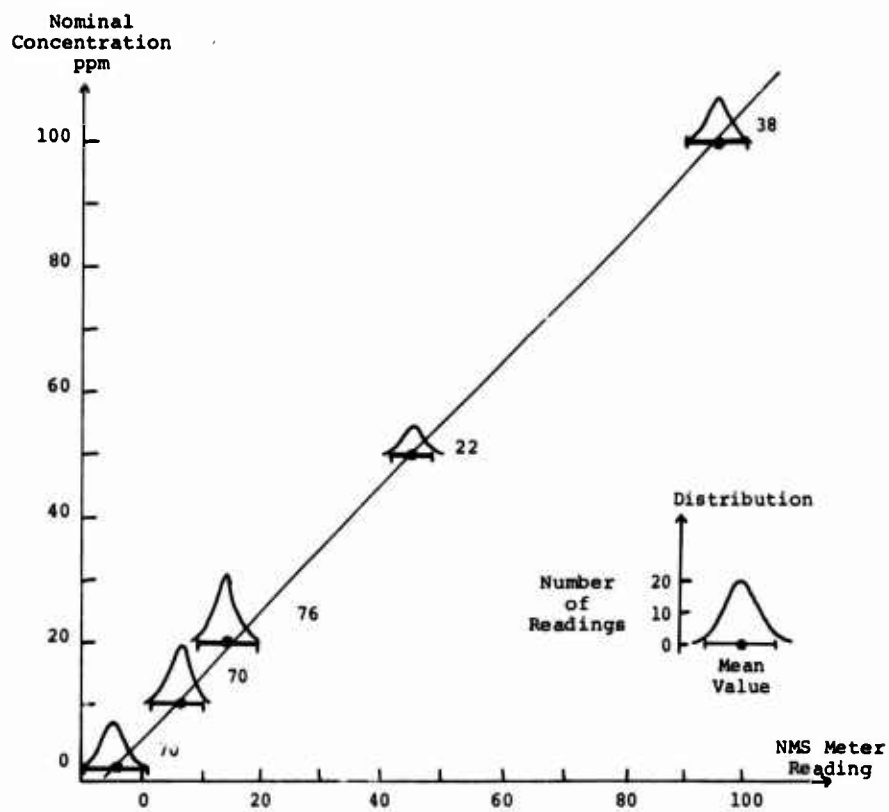


Figure 16. NMS Readout vs Concentration.

TABLE V. NMS READING VS CONCENTRATION
(12-MINUTE SAMPLE TIME)

0 ppm	70 readings	Mean = 3.9	$\sigma = 2.9$ ppm
Reading:	-11 -10 -9 -8 -7 -6 -5 -4 -3 -2 -1 0 1 2 3 4		
Frequency:	1 1 0 4 4 14 10 10 12 5 2 0 4 1 1 1		
10 ppm	70 readings	Mean = 5.9	$\sigma = 2.3$ ppm
Reading:	-1 0 1 2 3 4 5 6 7 8 9 10 11		
Frequency:	1 1 0 1 2 8 17 19 1 11 5 3 1		
20 ppm	76 readings	Mean = 14.0	$\sigma = 2.8$ ppm
Reading:	8 9 10 11 12 13 14 15 16 17 18 19 20 21		
Frequency:	1 3 4 6 9 23 3 9 9 3 3 1 1 1		
20 ppm	15 readings	Mean = 16.80	$\sigma = 3.4$ ppm
Reading:	12 13 14 15 16 17 18 19 20 21 22		
Frequency:	1 2 1 1 4 1 1 0 1 1 1		
50 ppm	22 readings	Mean = 45.2	$\sigma = 1.8$ ppm
Reading:	43 44 45 46 47 48 49		
Frequency:	4 4 6 0 7 0 1		
100 ppm	38 readings	Mean = 95.4	$\sigma = 2.8$ ppm
Reading:	91 92 93 94 95 96 97 98 99 100 101 102 103		
Frequency:	1 2 7 8 4 2 4 7 1 0 0 1 1		

TABLE VI. NMS READINGS VS CONCENTRATION
(45-SECOND SAMPLE TIME)

Date	Nominal Concentration	Number of Readings	Mean	Standard Deviation
2/1	20	20	20.6	6.3
	50	20	62.3	6.2
	100	20	98.6	5.5
2/8	10	8	4.5	5.2
	20	10	19.3	7.5
	50	11	51.1	4.9
	100	11	98.0	5.2
2/9	0	10	-6.4	4.7
	10	10	6.1	4.8
	10	10	9.0	5.3
	20	10	18.2	6.4
	50	10	47.2	6.4
	100	10	92.1	4.9
	100	10	93.7	7.5
2/18	0	10	0.0	7.6
	10	11	6.7	6.2
	20	10	20.9	5.8
	50	10	45.2	7.3
	100	10	93.2	5.8

energy resolution deteriorated from 18% (full width at half maximum - FWHM) to 22% (FWHM), but this was still adequate to detect the iron X-rays with no loss of NMS sensitivity.

The Pu²³⁸ source is classified by the manufacturer as suitable for industrial gauging applications. It meets the American National Standards Institute (ANSI) specification C-33232.⁽⁶⁾ In terms of temperature characteristics, the source remains intact between -71°F and 356°F. For vibration, the source has been subjected to 25-50 cps at 5 G and 50-500 cps at 10 G. It should be noted, however, that for radiological safety reasons the Pu²³⁸ source would not be used in airborne gauging applications. Krypton 85 sources have been used in airborne systems⁽¹⁾ and can be built to meet the required specifications.

4.0 DISCUSSION OF RESULTS

The yields and theoretical detection limits for the Pu^{238} , tritium, and Kr^{85} (Cu target) sources are presented in Table VII. Also included in the table are estimates made for a Kr^{85} source with a molybdenum (Mo) target. The two standard deviation detection limits have been normalized to a 12-minute count time. This count time could be reduced to 3 or 4 minutes by using stronger sources. For ground-based versions of the NMS, 50 millicuries of Pu^{238} could be used, while for airborne versions, 500 millicuries of Kr^{85} could be used. Gauges used in aircraft for measuring oil quantity use 500 millicuries of Kr^{85} , as specified in MIL-0-38338A.

In practice, the NMS using Pu^{238} successfully detected iron in oil with a two-standard-deviation detection limit of ± 5 ppm in the range 0-100 ppm. This is very close to the theoretical detection limit presented in Table VII. Tests showed that the NMS easily detected changes of 10 ppm. Other experimenters have sought to find small concentrations of metals in oil using XRF, but previously reported detection limits have all been above 50 ppm.^{(7) (8)} The present nucleonic metal sensor represents a substantial increase in sensitivity over previous efforts.

The use of rugged proportional counters and lightweight, maintenance-free sources, such as Pu^{238} or Kr^{85} , makes the NMS a practical instrument as well as a technically sophisticated one. The instrument must be calibrated and setup with great care, but once the NMS is ready for use, operation is extremely simple. Proportional counter tubes have been used as X-ray probes in several satellite programs⁽⁹⁾ and can easily be constructed to endure high-temperature, -vibratory environments. Both Pu^{238} and Kr^{85} sources are easy and economical to fabricate.

A great advantage of a nucleonic metal sensor based on X-ray fluorescence is its ability to detect and distinguish different metals. The NMS could be provided with several discriminator circuits, each set for a different metal; changing from metal to metal would be accomplished by a selector switch. Tests with the mixtures of iron and copper particles have shown that the NMS could be used to detect either iron or copper, or both, simultaneously.

Finally, the NMS has potential for wide application to any system where knowledge of metal concentration in oil is an important parameter.

TABLE VII. COMPARISON OF SOURCES TESTED					
Source	Strength	Fe X-rays (cps/ 10 ppm)	Back- ground (cps)	I _{Fe} /I Bkgd	2σ Detection Limits (12-minute sample)
Pu ²³⁸	10 mCi	1.33	44.5	0.030	± 3.8 ppm
H ³ (Tritium)	16 Ci	1.93	303	0.0064	± 6.7 ppm
Kr ⁸⁵ /Cu	120 mCi	2.91	893	0.0032	± 6.6 ppm
Kr ⁸⁵ /Mo*	120 mCi	2.00	225	0.0089	± 5.6 ppm
*Estimated yield					

5.0 POSSIBLE FUTURE PROGRAM CONSIDERATIONS

The present program has resulted in the successful development of a nucleonic metal sensor capable of detecting metals in oil to an accuracy of ± 5 ppm. The NMS can be constructed to endure the rigors of the airborne environment with little or no maintenance and excellent sensitivity and radiological safety factors.

There are two areas in which the NMS project might logically be continued. The first area involves a test program on helicopter gearboxes in ground based test cells, to determine the correlation between engine failure and concentration of metallic debris in oil. The second area involves design engineering on the NMS itself, to miniaturize the detector and mounting system for easy inclusion in airborne oil systems.

Detailed information about the rate of increase of metal in oil is not available at present. A ground based NMS designed to operate on a test stand gearbox could be used to study the relation between component failure and metal concentration. This study could include analysis of the lubricating oil by other techniques for comparison. A single electronics package could be used to control several sensors located in different areas of the transmission oil system. Examination of drawings supplied by the Directorate* suggests the sump and an area near the transmission oil pressure transmitter as possible locations for nucleonic metal sensors.

One extremely valuable piece of data would be the correlation between the concentrations of metal in solution and metal particles in the oil. This could be determined by examining the oil with the transmission operating and the metal particles circulating, and comparing this with readings obtained with filtered samples with particles removed. Information such as this would have direct correlation with the effectiveness of the off-line analysis such as the SOAP program.

A possible design for the airborne NMS is shown in Figure 17. Two detectors and two sources are used in this design to enable both sides of the oil flow path to be examined, and to effectively double the thickness of oil sampled. The outer container would be oil tight so that in the event of an overpressure rupturing the 10-mil beryllium windows, no oil would be lost from the lubricating system.

* Drawings of the UH-1 Transmission Oil System supplied by U. S. Army Air Mobility Research and Development Laboratory, Fort Eustis, Virginia.

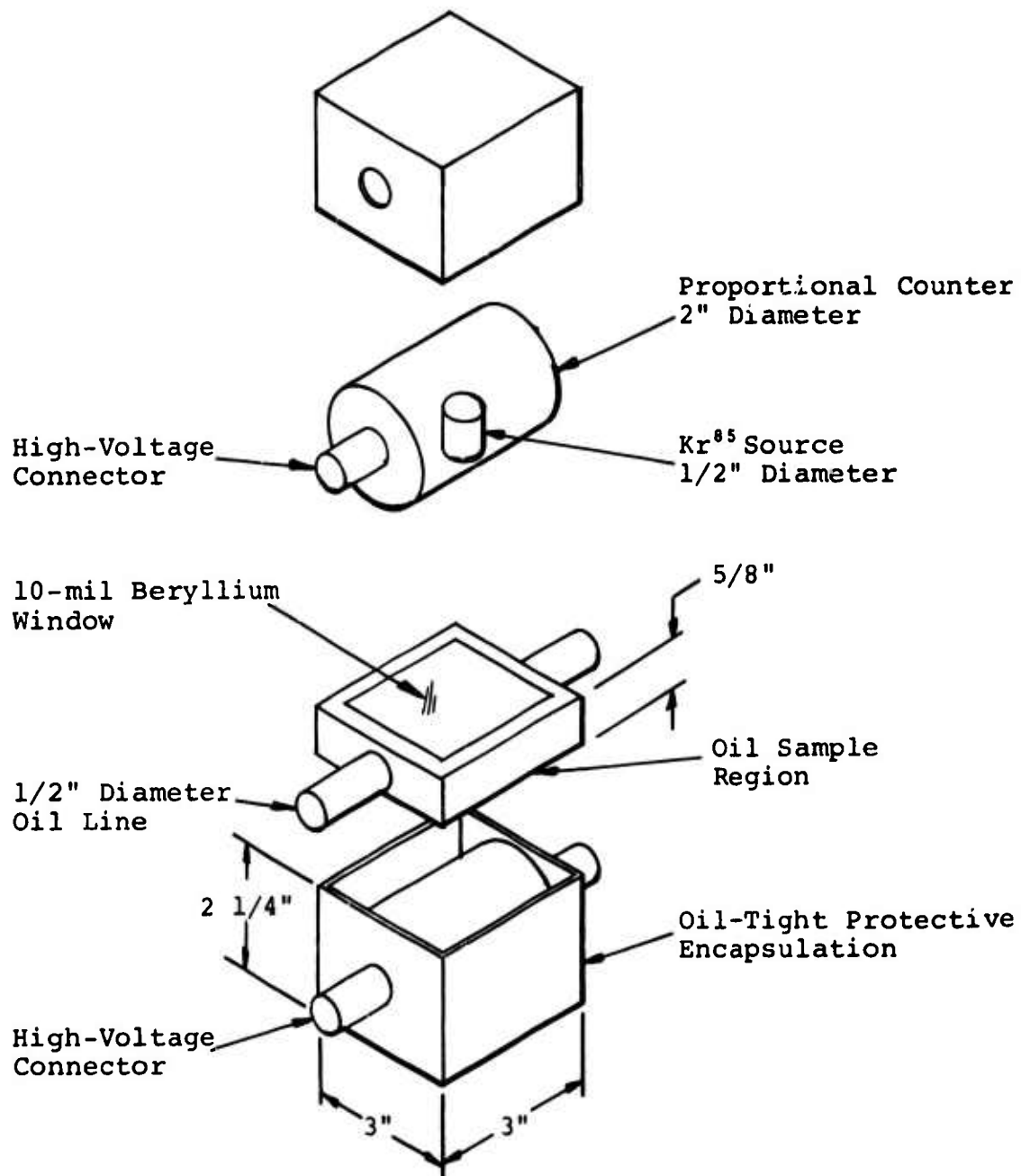


Figure 17. Airborne NMS Design.

There is another area where nucleonic technology might have application in measuring contamination in lubricating systems. This technique is outside the scope of the present contract, but it is mentioned here because it is related to the problem.

As a result of our work in air pollution measurement, our company has developed a beta gauging technique which is very effective in measuring the total mass of dirt accumulated on a filter, including metallic, as well as nonmetallic, material. Combining this technique with the XRF technique would provide an instrument which could measure the total amount of contamination present, identify and measure the total amount of heavy metals present, and, if desired, also measure the amount of metals in solution.

By using filters of selected sizes, all particles of any given size circulating in an oil or hydraulic oil system could be trapped and measured. It would be envisioned that this filter would be of small size, not part of the main filtering system, and located in the system to ensure maximum entrapment of all circulating particles.

This concept is presented as a possible approach to developing a total contamination sensing system.

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APPENDIX X-RAY ABSORPTION

Although the X-ray absorption method was not used as the final solution to the nucleonic metal sensor problem, XRA was shown to be capable of detecting metal in oil at the level of 10 ppm. The XRF and XRA programs shared many common features, particularly in the area of the electronics assembly. Valuable experience gained in the XRA studies was subsequently applied to the final configuration of the NMS using XRF. The middle third of the program was devoted primarily to X-ray absorption, and a brief summary of what was learned is presented in this appendix.

I. Theory

The principle of the X-ray absorption (XRA) method is shown in Figure 18. In clean oil, some of the photons from the X-ray source penetrate through the sample and are counted when they reach the detector. Other photons are absorbed by the oil molecules. The addition of a metallic contaminant to the oil does not change the absorption properties of the oil, but because the metal atoms absorb much more strongly than the oil atoms, a small increase in metal concentration will greatly increase the absorption of X-ray photons.

X-ray absorption is a well-known phenomenon. The intensity of a narrow beam of monoenergetic X-rays transmitted through a thickness x of absorber is given by

$$I = I_0 e^{-\mu \rho x} \quad (4)$$

Here I_0 is the detected beam intensity or count rate in the absence of an absorber, ρ is the absorber density (gm/cm^3), and μ the mass absorption coefficient (cm^2/gm) for x in cm. In a homogeneous metal-oil mixture, μ can be written

$$\mu = \mu_m p + \mu_o (1-p) \quad (5)$$

where p is the weight fraction of metal and μ_m and μ_o are absorption constants for metal and oil respectively. We can calculate the effect of a change in metal concentration, Δp , on transmitted beam intensity:

$$\Delta I = -I (\mu_m - \mu_o) \rho x \Delta p \quad (6)$$

We wish to maximize this change compared to the statistical uncertainty $\Delta I_s = \sqrt{I}$. We define a figure of merit

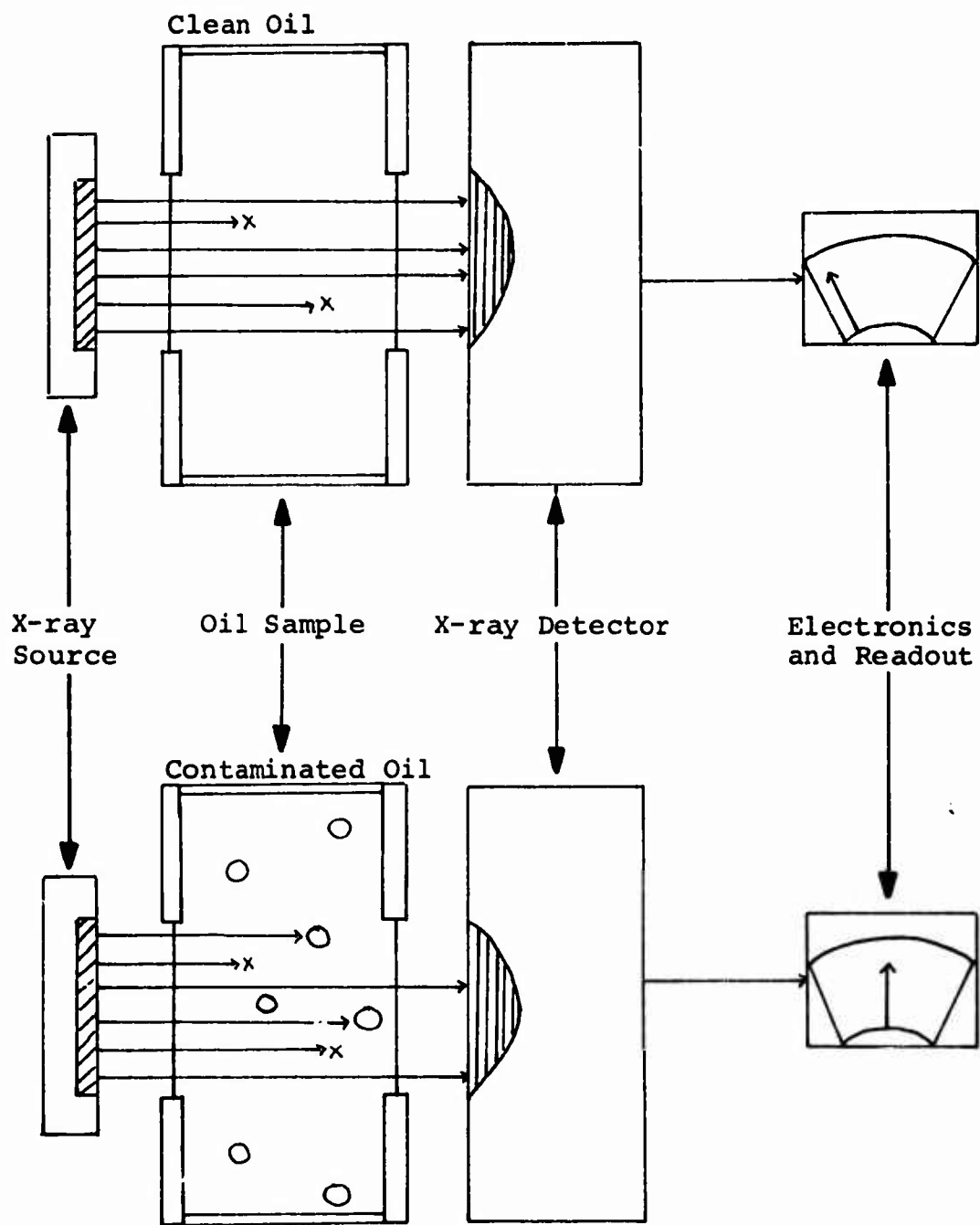


Figure 18. X-Ray Absorption System.

$F = \Delta I / \Delta I_s$ and maximize F with respect to x . The maximum value of F occurs for $x = 2/\rho\mu$.

We again approximate

$$\mu_{\text{oil}} \approx \mu_{\text{carbon}}$$

Figure 2 shows that

$$\mu_{\text{iron}} > 70 \mu_{\text{carbon}}$$

for low-energy X-rays; but for

$$p < 10^{-3}$$

equation (5) becomes

$$\mu \approx \mu_{\text{carbon}}$$

Substituting this into equation (6) with

$$\rho x \approx 2/\mu_{\text{carbon}}$$

and

$$(\mu_m - \mu_o) \approx \mu_{\text{iron}}$$

we find

$$\Delta I = -I 2 \frac{\mu_{\text{iron}}}{\mu_{\text{carbon}}} \Delta p$$

Table VIII lists the ratio

$$\mu_{\text{iron}} / \mu_{\text{carbon}}$$

the percentage change in count rate for

$$\Delta p = 10^{-3} \text{ (10 ppm)}$$

and the optimum thickness for a wide range of X-ray energies. In calculating x , we have used a value of

$$\rho = 0.95 \text{ gm/cm}^3$$

for the oil. Also shown in the table is the number of counts required to make the statistical fluctuations the equivalent of 10 ppm or less. This information indicates what source strength will be needed for an XRA version of the nucleonic metal sensor.

The above calculations have involved many simplifying assumptions, such as monoenergetic, well-collimated X-ray beams and equal photon path lengths through the oil. In practice, the sensitivity of the NMS may be less than

TABLE VIII. X-RAY ABSORPTION PARAMETERS VS INCIDENT ENERGY				
E (keV)	$\mu_{\text{iron}}/\mu_{\text{carbon}}$	$\Delta I/I$ at 10 ppm	x (cm)	Counts
6	8.1	0.02 %	0.2	3×10^7
8	72	0.14 %	0.5	5×10^5
10	79	0.16 %	1.0	4×10^5
15	78	0.16 %	2.8	4×10^5
20	62	0.12 %	5.1	7×10^5
30	33	0.07 %	8.1	2×10^6
40	17	0.03 %	10	1×10^7
50	10	0.02 %	11	3×10^7
100	2.4	0.005%	14	4×10^8

calculated. However, we expect roughly a 0.2% decrease in count rate for every 10 ppm of iron added to the clean oil.

The effect of temperature changes on count rate can also be estimated. As the temperature increases by an amount ΔT , the oil density will change due to volume expansion. We have

$$V = V_o (1 + \alpha \Delta T) \quad (7)$$

$$\rho = \rho_o / (1 + \alpha \Delta T) \quad (8)$$

Here V_o is the original volume, ρ_o the original density, and α the volume expansion coefficient. For most oils,

$$\alpha \approx 0.3 \times 10^{-3} / ^\circ\text{F}$$

at 60°F . Inserting equation (8) into equation (4) and differentiating with respect to temperature change, we find that the count-rate change due to temperature change is

$$\Delta I_T = I \mu \rho_o \times \frac{\alpha \Delta T}{(1 + \alpha \Delta T)} \quad (9)$$

But we have chosen

$$\mu \rho_o \times = 2$$

and for

$$\alpha \Delta T \ll 1$$

we find the percentage change in count rate

$$\Delta I_T / I = 0.05\% / ^\circ\text{F}$$

Every change of 4°F causes an effect which is the same as changing the metal concentration by 10 ppm. Thus, temperature compensation is of the utmost importance in applying XRA as a tool for locating metal in oil at the level of 10 ppm.

II. Experimental Procedures

The X-ray absorption scheme shown in Figure 18 detects contamination by measuring a change in the number of photons transmitted through the oil. Table VIII shows that this change is maximum for 8-20 keV photons. The Kr^{85} and tritium sources discussed previously were also tested for use in X-ray absorption. The high-energy bremsstrahlung spectrum from the Kr^{85} source reduced the

sensitivity of this source in the XRA configuration. In addition, the beryllium window required to contain the beta particles reduced the output of 8 keV copper X-rays. Tritium was selected as an alternative source. To increase the sensitivity of the NMS, a 1-mil-thick copper absorption edge filter was used to filter out the high-energy portion of the tritium bremsstrahlung spectrum, and the oil sample itself acted as a filter for the low-energy portion of the spectrum.

Energy resolution is not a prime requisite for the detector in an XRA system, and avalanche-type detectors were investigated in detail. The avalanche detector is the solid-state analog of the gas-filled proportional counter. The detectors purchased proved unsatisfactory for use in the NMS; the junction depth was variable, causing the amplitude of the output pulses to fluctuate. In addition, the detectors were not capable of operating at elevated temperatures. An argon-filled proportional counter was eventually selected for use in the XRA system. By making use of the energy discrimination capability of the proportional counter system, the sensitivity of the instrument could be increased.

A measuring head was constructed which held the source, detector, oil sample, platinum resistance thermometer, and heaters. The source was mounted on a threaded insert so the thickness of the oil sample could be varied. The optimum thickness proved to be 0.16 inch. This was a drawback for the XRA system since a constriction would be required in the oil flow path.

During the XRA program, the basic electronics unit was assembled and the concept of temperature compensation was first implemented. With changes in the D/AC circuit, timer, and scaler circuits, this unit was later used for XRF.

In order to reduce statistical fluctuations to the level of ± 5 ppm, measurement times of 22 minutes were required. During this time the NMS scaler accumulated 2.7×10^6 counts, at a rate of approximately 2000 cps. At 500 ppm, the measured decrease in count rate was 9.4%. At lower concentrations, the decreases were: 100 ppm - 2.2%; 20 ppm - 0.39%; and 10 ppm - 0.17%. Temperature changes of $\pm 30^\circ\text{F}$ caused fluctuations of $\pm 0.36\%$ in the number of counts accumulated. This reduced the sensitivity of the NMS to ± 20 ppm.

Several proposals were advanced to perform temperature

compensation. The first one involved using an arbitrary function generator to vary counting time with temperature. This did not prove to be feasible because of the long counting times required for each measurement. A second approach was to keep the measuring head at an elevated temperature. This temperature would be maintained by a heating circuit. The heating circuit in turn was to be controlled by a feedback signal from the temperature sensing electronics. Temperature compensation appeared feasible, but difficult. It was decided at this point in the program to expend the balance of the time and funds on the XRF work.